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Indian Standard

SPECIFICATION FOR DISODIUM PHOSPHATE, DODECAHYDRATE

(Second Revision)

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Indian Standard

SPECIFICATION FOR DISODIUM PHOSPHATE, DODECAHYDRATE

(Second Revision)

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Indian Standard
SPECIFICATION FOR DISODIUM PHOSPHATE, DODECAHYDRATE
(*Second Revision*)

0. FOREWORD

0.1 This Indian Standard (Second Revision) was adopted by the Indian Standards Institution on 27 July 1984, after the draft finalized by the Inorganic Chemicals (Misc) Sectional Committee had been approved by the Chemical Division Council.

0.2 This standard was first revised in 1965 amalgamating IS : 572-1954* and IS : 566-1954†, when the requirement of phosphates (as P_2O_5) prescribed in IS : 572-1954* had been dropped. A reference had also been made to 'Modified gutzeit method of test for arsenic' (IS : 2088-1962) for the determination of arsenic, and changes in the method of sampling were also effected.

0.3 This standard has been revised again in the light of the developments made by the industry. In this revision, only two grades have been specified, namely, technical and analytical reagent grades. Pharmaceutical grade has been deleted as this has already been covered in Indian Pharmacopocia. A new requirement of phosphates (as P_2O_5) with quionoline phospho-molybdate method of test has been incorporated and the method for the determination of disodium phosphate content has also been revised.

0.4 The single largest use of disodium phosphate is as an emulsifier for pasteurised processed cheese. Its other uses in food industry are in ham curing, starch processing and as an ingredient in instant cereals and evaporated milk. It is also used in the preparation of ceramic glazes and enamel in leather tanning, textile dyeing, in pharmaceuticals, water treatment and detergents.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960‡. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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*Specification for disodium phosphate, dodecahydrate, technical.

†Specification for disodium phosphate, dodecahydrate, pharmaceutical and analytical reagent.

‡Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for disodium phosphate, dodecahydrate (Na_2HPO_4 12 H_2O).

2. GRADES

2.1 The material shall be of the following two grades:

a) Technical (TECH), and

b) Analytical Reagent (AR).

2.2 The technical grade is generally used for boiler water conditioning, textile processing and in the manufacture of detergents, and in leather tanning and ceramic industry.

3. REQUIREMENTS

3.1 Description — The material shall be in the form of colourless and odourless crystals or granules. It readily loses water on exposure to air at ordinary temperature.

3.2 The material shall comply with the requirements given in Table 1 when tested according to the methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given in col 5 of the table.

4. PACKING AND MARKING

4.1 Packing

4.1.1 The technical grade of the material shall be packed in polyethylene lined jute bags or as agreed to between the purchaser and the supplier.

4.1.2 The analytical reagent grade of the material shall be packed in glass bottles with moistureproof glass or plastic stoppers. The bottles shall be labelled with full analytical data of the characteristics prescribed under Table 1.

4.2 Marking

4.2.1 The container shall be securely closed and marked with the name of the manufacturer, the grade and mass of the material in the container, recognized trade-mark, if any, and the year of manufacture of the material.

4.2.2 The material may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standard Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

TABLE 1 REQUIREMENTS FOR DISODIUM PHOSPHATE, DODECAHYDRATE

(*Clause* 3.2)

SL No.	CHARACTERISTIC	REQUIREMENT OF GRADE		METHOD OF TEST (REF TO CL NO. IN APPENDIX A)
		TECH	AR	
(1)	(2)	(3)	(4)	(5)
i)	Loss on drying, percent by mass	57·0 to 61·0	57·0 to 61·0	A-2
ii)	Disodium phosphate content (as Na_2HPO_4) on dry basis, percent by mass, *Min*	98·0	99·0	A-3
iii)	Matter insoluble in water, percent by mass, *Max*	0·25	To pass test	A-4
iv)	Chlorides (as Cl), percent by mass, *Max*	—	0·002	A-5
v)	Sulphates (as SO_4), percent by mass, *Max*	—	0·010	A-6
vi)	Soluble iron compounds (as Fe), percent by mass, *Max*	0·01	0·0005 (5 parts per million)	A-7
vii)	Heavy metals (as Pb), percent by mass, *Max*	—	do	A-8
viii)	*p*H (2·0 percent (*m*/*v*) solution) at 27°C	—	9·0 to 9·2	A-9
ix)	Arsenic (as As_2O_3) parts per million, *Max*	—	2	A-10
x)	Carbonates	—	To pass test	A-11
xi)	Calcium and magnesium	—	—	A-12
xii)	Phosphates (as P_2O_5), percent by mass, *Min*	49·0	49·5	A-13*

*This shall be on dry basis.

5. SAMPLING

5.1 The method of drawing representative samples of the material and the criteria for conformity shall be as prescribed in Appendix B.

APPENDIX A

(*Clause* 3.2)

METHOD OF TEST FOR DISODIUM PHOSPHATE, DODECAHYDRATE

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF LOSS ON DRYING

A-2.1 Procedure — Weigh accurately about 10 g of the material in a tared porcelain basin of 6-8 cm diameter and 1-3 cm depth. Dry the contents of the basin to constant mass at 130 ± 2°C.

A-2.2 Calculation

$$\text{Loss on drying, percent by mass} = 100\frac{M_1}{M_2}$$

where

M_1 = loss in mass in g, and

M_2 = mass in g of the material taken for the test.

A-3. DETERMINATION OF DISODIUM PHOSPHATE (Na_2HPO_4)

A-3.0 Principle — Disodium phosphate content is determined volumetrically by treating solution with excess hydrochloric acid and then by titration with standardized sodium hydroxide solution.

*Specification for water for general laboratory use (*second revision*).

A-3.1 Apparatus

A-3.1.1 *Beaker* — 250 ml capacity, marked at 100-ml level.

A-3-1.2 *pH Meter*

A-3.2 Reagents

A-3.2.1 *Dilute Hydrochloric Acid* — 5N.

A-3.2.2 *Mixed Indicator Solution* — Mix equal volumes of bromocresol green (0·1 percent in water or 20 percent alcohol), phenolphthalein (0·5 percent in 60 percent alcohol) and absolute alcohol.

A-3.2.3 *Sodium Hydroxide Solution* — 5N.

A-3.2.4 *Standardized Sodium Hydroxide Solution* — 0·1N.

A-3.3 Procedure — Weigh accurately about 1 g of the material dried as under **A-2.1** and dissolve in water and dilute it to 100 ml in volumtrict flask and shake well to mix. Pipette out 50 ml into a 250 ml beakereand shake well to mix. Pipette out 50 ml into a 250 ml beaker marke d a 100 ml level. Add 15 ml dilute hydrochloric acid and dilute to 100 ml. Introduce a boiling rod, cover with a watch glass and boil gently for 30 minutes. Add 0·5 ml of the mixed indicator, and neutralize the solution with sodium hydroxide solution (5N) to a blue-green colour. Add dilute hy drochloric acid (0·5 N) until indicator changes to yellow (about 0·5 ml is usually needed).

A-3.3.1 Dilute to 100 ml, if necessary, and boil for 15 minutes to expel carbon dioxide. Cool to room temperature. Wash and remove cover glass and wash boiling rod and wall of the beaker. Dilute to 100 ml. Place the beaker on the *p*H-meter assembly. With thorough stirring (preferably mechnical) add standard sodium hydroxide solution (0·1N) until *p*H reaches 4·1.

A-3.3.2 Titrate further to *p*H 8·8 with the same standard sodium hydroxide solution (0·1 N). The final volume shall be about 150 ml; hence, excessive rinsing of the burette tip, electrodes, etc, should be avoided.

A-3.4 Standardization of N/10 Sodium Hydroxide Solution — Analyse a sample of pure anhydrous tetrasodium pyrophosphate ($Na_4P_2O_7$) by proceeding exactly as above except for accurately weighing 3·8 g sample, diluting it to 500 ml and pipetting out 75 ml of diluted solution.

$$F = \frac{53{\cdot}38 \times M}{4{\cdot}732 \times T}$$

where M = mass in g of pure tetrasodium pyrophosphate

if pure anhydrous tetrasodium pyrophosphate is not available, pure potassium dihydrogen orthophosphate crystals may be used in the same manner for standardization of N/10 sodium hydroxide solution,

$$\text{then } F = \frac{52{\cdot}17 \times M}{4{\cdot}732 \times T}$$

where

M = mass in g of potassium dihydrogen orthophosphate taken.

A-3.5 Calculation

$$\text{Disodium phosphate (as } Na_2HPO_4\text{), percent by mass} = \frac{T \times F}{M} \times 2{\cdot}84$$

where

T = difference in volume in ml in sodium hydroxide solution required in **A-3.3.1** and **A-3.3.2**,

F = factor of N/10 sodium hydroxide solution (*see* **A-3.5**), and

M = mass in g of the material taken for the test

A-4. DETERMINATION OF MATTER INSOLUBLE IN WATER

A-4.1 For Technical Grade

A-4.1.1 *Procedure* — Weigh accurately about 10 g of the material and dissolve in about 150 ml of water. Filter the residue, if any, through a tared filter paper or a sintered glass crucible (G No. 4) or a Gooch crucible. Wash the residue thoroughly with water till it is free from all soluble compounds and dry to constant mass at 105 to 110°C. Cool in a desiccator and weigh.

A-4.1.1.1 *Calculation* — Express the weight of the residue as percentage of the mass of the material taken for the test.

A-4.2 For Analytical Reagent Grade

A-4.2.1 *Procedure* — Weigh about 10 g of the material and dissolve in 100 ml of water at 27 ± 2°C. Shake well and allow to stand for 15 minutes.

A-4.2.1.1 The material shall pass the requirement of the test if there is no sediment.

A-5. TEST FOR CHLORIDES (Cl)

A-5.0 Principle — Chlorides are determined by comparing the opalescence produced with silver nitrate solution against that of a standard solution of chloride in a Nessler cylinder.

A-5.1 Apparatus

A-5.1.1 *Nessler Cylinder* — 50 ml capacity.

A-5.2 Reagents

A-5.2.1 *Concentrated Nitric Acid — See* IS : 264-1976*.

A-5.2.2 *Standard Chloride Solution* — Dissolve 1·649 g of sodium chloride in water and make up the volume to 1 000 ml. Pipette out 10 ml of the solution, dilute with water and make up the volume to 100 ml. One millilitre of this solution contains 0·1 mg of chloride (as Cl).

A-5.2.3 *Silver Nitrate Solution* — Approximately 5 percent (*m/v*).

A-5.3 Procedure for the Analytical Reagent Grade — Weigh 5·0 g of the material and dissolve in 20 ml of water. Transfer the solution completely into a Nessler cylinder and proceed as under A-5.3 with the modification that in the control test one millilitre of standard chloride solution shall be used.

A-6. TEST FOR SULPHATES (SO_4)

A-6.1 For Analytical Reagent Grade

A-6.1.1 *Procedure* — Dissolve 2·50 g of the material in 50 ml of water. Add 2·5 ml of dilute hydrochloric acid and 2 ml of barium chloride solution. Mix thoroughly and allow the reaction mixture to stand for 2 hours. Note if any turbidity or precipitate is formed at the end of this period.

A-6.1.1.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if no turbidity or precipitate is produced.

A-7. DETERMINATION OF SOLUBLE IRON COMPOUNDS (Fe)

A-7.0 Principle — Soluble iron compounds and heavy metals are determined calorimetrically by visual comparison of colour in Nessler cylinders.

A-7.1 Apparatus

A-7.1.1 *Nessler Cylinder* — 50 ml capacity.

A-7.2 Reagents

A-7.2.1 *Concentrated Hydrochloric Acid — See* IS : 265-1976†.

A-7.2.2 *Ammonium Persulphate*

A-7.2.3 *Butanolic Potassium Thiocyanate* — Dissolve 10 g of potassium thiocyanate in 10 ml of water. Add sufficient *n*-butanol to make up to 100 ml and shake vigorously until the solution is clear.

*Specification for nitric acid (*second revision*).

†Specification for hydrochloride acid (*second revision*).

A-7.2.4 *Standard Iron Solution* — Dissolve 0·702 g of ferrous ammonium sulphate [$FeSO_4.(NH_4)_2SO_4.6H_2O$] in 10 ml of dilute sulphuric acid (10 percent by volume) and dilute with water to make up the volume to 1 000 ml. Pipette out 10 ml of this solution and dilute with water to make up the volume to 100 ml. One millilitre of this solution is equivalent to 0·01 mg of iron (as Fe).

A-7.3 Procedure for Analytical Reagent Grade — Dissolve 2·00 g of the material in 20 ml of water. Add one millilitre of hydrochloric acid, 30 mg of ammonium persulphate and 15 ml of butanolic potassium thiocyanate solution. Make up the solution to 50 ml. Shake vigorously for 30 seconds and allow the liquids to separate. Carry out a control test with one millilitre of standard iron solution in place of the sample and the same quantities of other reagents in the same total volume of the reaction mixture. Compare the colour of the butanol layer in the two sets.

A-7.3.1 The limits prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced in the test with the material is not greater than that produced in the control test.

A-7.4 Procedure for Technical Grade — Dissolve 1·00 g of the material in water and make up the volume to 100 ml. Transfer 10 ml of this solution into a Nessler cylinder and dilute it with 30 ml of water. Add one millilitre of concentrated hydrochloric acid, 30 mg of ammonium persulphate and 15 ml of butanolic potassium thiocyanate. Make up the solution to 15 ml. Shake vigorously for 30 seconds and allow the butanol layer to separate. Carry out a control test in the second Nessler cylinder using one millilitre of standard iron solution in place of the material and same quantities of other reagents in the same total volume of reaction mixture. Compare the colour produced in the two cylinders after two minutes.

A-7.4.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the colour produced in the test with the material is not deeper than that produced in the control test.

A-8. DETERMINATION OF HEAVY METALS (Pb)

A-8.0 Principle — Heavy metals are determined by comparing the colour produced by the material with sodium sulphide solution against that of a standard lead solution in Nessler cylinders.

A-8.1 Apparatus

A-8.1.1 *Nessler Cylinders* — 50 ml capacity.

A-8.2 Reagents

A-8.2.1 *Dilute Hydrochloric Acid* — 5 N (approx).

A-8.2.2 *Acetic Acid* — Approximately 33 percent (*m/m*).

A-8.2.3 *Dilute Ammonium Hydroxide Solution* — Approximately 10 percent (*m/m*).

A-8.2.4 *Hydrogen Peroxide Solution* — Approximately 6 percent (*m/v*).

A-8.2.5 *Potassium Cyanide Solution* — Dissolve 10·0 g of potassium cyanide in 90 ml of water, add 2 ml of hydrogen peroxide solution, allow to stand for 24 hours and make up to 100 ml with water.

A-8.2.6 *Concentrated Nitric Acid — See* IS : 264-1976*.

A-8.2.7 *Standard Lead Solution* — Dissolve 1·60 g of lead nitrate [$Pb(NO_3)_2$] in water and make up the solution to 1 000 ml. Pipette out 10 ml of the solution and dilute again to 1 000 ml with water. One millilitre of this solution contains 0·01 mg of lead (as Pb). Final dilution shall be made afresh.

A-8.2.8 *Sodium Sulphide Solution* — Approximately 10 percent (*m/v*).

A-8.3 Procedure — Weigh 7·0 g of the material and dissolve in 15 ml of dilute hydrochloric acid and heat to boiling. Cool and transfer this solution completely into a Nessler cylinder and add 5 ml of acetic acid. Make it alkaline to litmus by gradual addition of dilute ammonium hydroxide solution and then add one millilitre of potassium cyanide solution. Carry out a control test in the other Nessler cylinder using 2·0 g of the material, 2·5 ml of standard lead solution and the same quantities of other reagents. Filter both the solutions, if they are turbid, if the colours of the solutions differ, equalize them by the addition of a few drops of a highly diluted solution of burnt sugar or any other non-reactive substance. Dilute both the solutions with water and make up the volume to 50 ml. Add 2 drops of sodium sulphide solution. Mix thoroughly and compare the colours develloped in the two cylinders.

A-8.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced in the test with the material is not greater than that produced in the control test.

A-9. DETERMINATION OF *p*H

A-9.1 Reagent

A-9.1.1 *Thymol Blue Indicator Solution (Alkali Range)*—*p*H 8·0 to 9·6 — Colour change yellow to blue — Dissolve 0·04 g of thymol blue in 50 ml of rectified spirit (conforming to IS : 323-1959†) and dilute to 100 ml with water.

A-9.2 Test Temperature — The test shall be carried out at 27°C.

*Specification for nitric acid (*second revision*).

†Specification for rectified spirit (*revised*).

A-9.3 Procedure — In the hard glass tube, place 10 ml of the solution prepared by dissolving 2·00 g of the material in 100 ml of carbon dioxide free water. Determine the *p*H of this solution using 5 drops of thymol blue indicator solution. Compare the colour produced with that of a series of buffer tubes of known *p*H each containing the same proportion of the indicator. Standard calibrated glass discs may also be used for comparison.

A-9.3.1 The *p*H may also be measured with glass electrodes.

A-9.4 Report as *p*H, the *p*H of the buffer solution which matches with that of the material.

A-10. TEST FOR ARSENIC (As_2O_3)

A-10.1 Dissolve 1·00 g of the material in 15 ml of water and carry out the test for arsenic as prescribed in IS : 2088-1971*, using for comparison a stain obtained with 0·002 mg of arsenic trioxide (as As_2O_3) in case of analytical reagent grade.

A-10.2 The limit prescribed in Table 1 shall be taken as not having been exceeded if the length of the stain as well as the intensity of its colour produced in the test with material are not greater than those of the stain produced with the standard arsenic trioxide solution.

A-11. TEST FOR CARBONATES

A-11.1 Reagent

A-11.1.1 *Dilute Hydrochloric Acid* — Approximately 5 N.

A-11.2 Procedure — Dissolve 5·0 g of the material in 50 ml of boiling water. Add 10 ml of dilute hydrochloric acid to this hot solution. The material shall be taken to have satisfied the requirement of the test if no effervescence is produced.

A-12. TEST FOR CALCIUM AND MAGNESIUM

A-12.1 Procedure — Dissolve 2·0 g of the material in 50 ml of water, add three millilitre of dilute ammonium hydroxide solution (approximately 5N). Set aside for five minutes and note if any turbidity is produced. The material shall be regarded to have passed the test if no turbidity is produced.

A-13. TEST FOR PHOSPHATES (P_2O_5)

A-13.1 The quinoline phosphomolybdate method as given in 4 of IS : 5305-1969† shall be followed for the determination of phosphates after dissolving about 1 g of the (dried) material (**A-2.1**) in 100 ml of water.

*Methods for determination of arsenic (*first revision*).

†Methods for volumetric determination of phosphorus.

APPENDIX B

(*Clause* 5.1)

SAMPLING OF DISODIUM PHOSPHATE, DODECAHYDRATE

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling samples, the following precautions and directions shall be observed.

B-1.1 Samples shall not be taken in an exposed place.

B-1.2 The sampling instrument shall be clean and dry when used.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 To draw a representative sample the contents of each package selected for the sampling shall be mixed as thoroughly as possible by suitable means.

B-1.5 The samples shall be placed in clean, dry and air-tight glass or any other suitable containers on which the material has no chemical action.

B-1.6 The sample containers shall be of such a size that they are almost completely filled by the samples.

B-1.7 Each sample container shall be suitably stoppered and sealed air-tight after filling and marked with full particulars of the material (*see* **4.2**) and the date of sampling.

B-1.8 Samples shall be stored in a cool and dry place.

B-2. SCALE OF SAMPLING

B-2.1 Lot — All the packages in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture, the batches shall be marked separately and the groups of packages in each batch shall constitute separate lots.

B-2.2 For ascertaining the conformity of the material in a lot to the requirements of this specification, samples shall be tested for each lot separately. The number of packages to be selected at random from lot of different sizes shall be in accordance with Table 2.

TABLE 2 NUMBER OF PACKAGES TO BE SELECTED

(*Clause* B-2.2)

Lot Size	Sample Size
N	n
(1)	(2)
Up to 50	3
51 ,, 300	4
301 ,, 500	5
501 ,, 1 000	7
1 001 and above	10

B-2.3 In order to ensure randomness of selection, use shall be made of random number table, but if such a table is not available the following procedure may be adopted:

Starting from any package, count them as 1, 2, 3,..... up to r and so on where r is the integral part of N/n (N being the lot size and n the sample size). Every rth package thus counted shall be drawn to constitute the sample.

B-3. INDIVIDUAL SAMPLES AND COMPOSITE SAMPLES

B-3.1 From each of the packages selected according to **B-2.3**, a representative portion of the material shall be drawn with the help of appropriate sampling instrument. The amount so withdrawn from each package shall be sufficient for carrying out all the tests specified under **B-4** and shall constitute the individual sample.

B-3.2 From each of the individual samples, a small but equal quantity of the material shall be taken and thoroughly mixed to constitute a composite sample.

B-3.3 Each of the individual samples and the composite sample shall be transferred to separate bottles and labelled with full identification particulars.

B-4. NUMBER OF TESTS

B-4.1 Test for determination of loss on drying, disodium phosphate content and matter insoluble in water for technical grade and loss on drying, disodium phosphate content for AR grade shall be carried out on each of the individual samples.

B-4.2 Tests for the determination of the remaining characteristics shall be performed on the composite sample only.

B-5. CRITERIA FOR CONFORMITY

B-5.1 For Individual Samples — For those characteristics which are tested on individual samples, the mean and the range of test results shall be computed as follows:

$$\text{Mean } \bar{x} = \frac{\text{Sum of individual test results}}{\text{Number of test results}}$$

Range (R) = difference between the maximum and the minimum values of test results.

For declaring the conformity of the lot:

$\bar{x} + 0{\cdot}6\,R$ shall be less than or equal to the maximum specified requirements, and

$\bar{x} - 0{\cdot}6\,R$ shall be greater than or equal to the minimum specified requirements.

B-5.2 For Composite Sample — For declaring the conformity of the lot to the requirements of all the characteristics tested on the composite sample the test results shall satisfy the corresponding specified requirements.

(*Continued from page* 2)

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